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**EFFECT OF SPUTTERING PARAMETERS ON
TANTALUM COATINGS FOR GUN BORE APPLICATIONS**

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13. ABSTRACT (Maximum 200 words) Tantalum offers a number of attractive properties for gun bore coating applications, including a high melting temperature, high ductility, and an environmentally friendly deposition method. However, vapor-deposited tantalum can appear in both the characteristic body-centered-cubic phase found in the bulk material, and in a very brittle and less desirable "beta" phase. Presence of the beta phase in bore coatings is considered undesirable because of its brittleness and resulting failure as the coating is stressed. A high-rate triode sputtering system with a cylindrical coating geometry was used to produce thick tantalum coatings on 4340 steel, smooth bore cylindrical substrates. A systematic series of tests was performed to evaluate the effects of sputtering gas species (argon, krypton, xenon) and substrate temperature (100° to 300°C) during deposition on the phase and microstructure of the coatings. Heavier sputtering gases and higher substrate temperatures were found to promote the formation of body-centered-cubic phase tantalum coatings. Use of a movable target assembly was shown to promote the production of dense, single-phase tantalum coatings.					
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INTRODUCTION

The bore surfaces of many medium and large caliber guns are coated with chromium metal to increase the service life of the tubes, which are subject to erosive and corrosive wear. Current practice uses wet chemical electrodeposition to apply the chromium over the tempered steel used to form the tube. As environmental regulations become increasingly stringent and service requirements for the coatings become more demanding with use of higher energy propellants, alternative coating technologies and materials are being evaluated for this application. Metallic tantalum is one material that has received attention as a potential replacement for chromium in gun tubes. Bulk tantalum is highly refractory (melting point = 2996°C), is chemically resistant to propellant gases, has a relatively low thermal conductivity, and is much more ductile than chromium. Various methods have been or are being evaluated for the production of protective tantalum coatings on gun bore surfaces (refs 1-5).

Vapor-deposited tantalum coatings are often found to contain one or both of two distinct crystalline phases (ref 6). A body-centered-cubic (bcc) alpha phase is characteristic of bulk tantalum and has physical properties making it desirable for gun tube coating applications. A second metastable phase referred to as beta-tantalum has a tetragonal structure and was first identified in sputtered tantalum films (ref 7). Although the physical properties of beta-tantalum have not been well characterized, it is known to be thermally unstable, and reverts to the bcc phase at temperatures above 750°C. Many vapor-deposited tantalum coatings have been found to have both phases present, with the beta phase often appearing in conical growths and in interfacial layers that transition into the bcc phase as the coating grows. A number of studies have been previously undertaken in efforts to relate various sputtering conditions to the formation of one phase over the other (refs 7-20). However, few of these prior studies have dealt with high-rate depositions such as those that will be required to produce the relatively thick coatings (~100 µm) needed for gun bore applications.

We have undertaken an investigation of the tantalum-phase formation and distribution in thick films (~100 µm) produced using a cylindrical-geometry, high-rate triode sputtering apparatus. This report summarizes the results of investigations into the effects of sputtering gas species and substrate temperature on the phase composition and distribution in the thick sputtered tantalum coatings. Prior work has shown that the use of a niobium interlayer promotes the formation of bcc tantalum on gun steel (refs 4,21). Determination of coating parameters that allow production of the bcc phase directly on steel surfaces will eliminate the cost and other complications resulting from the intermediate niobium deposit.

Prior studies relating phase characteristics of sputtered tantalum to deposition conditions have almost exclusively utilized argon and doped argon as the sputtering gas, although the use of krypton and xenon has been reported by a few researchers (refs 4,18-21). Economic considerations are generally thought to favor the use of argon over the heavier and more expensive noble gases, particularly for industrial sputtering processes. However, the higher sputtering yields and resulting shorter run times to produce comparable thicknesses of coating may partially offset the higher cost of krypton or xenon where thick coatings are required, as in gun tube applications. Similarly, the effect of substrate temperature on tantalum coating phase characteristics has been studied (refs 16,17). However, these prior investigations have involved flat-plate geometries, different substrate materials, and argon as the sputtering gas. We felt it valid to evaluate this variable for thick tantalum coatings applied to steel substrates in the cylindrical geometry and using heavier sputtering gases. The temperatures evaluated in this

study (100° to 300°C) likely fall within the range of practical steel gun tube coating temperatures, which are limited on the upper end because of prestress treatments commonly applied to the tubes prior to bore coating processes.

EXPERIMENTAL METHOD

Coating Deposition

A cylindrical-geometry, triode sputtering system similar to one described in a previous publication (ref 4) was used to produce tantalum coatings on the inner surfaces of 25-mm inner diameter (ID) by 42-mm outer diameter (OD) 4340 steel right circular cylinders. The vacuum chamber body used for the current work was sized to accommodate substrate cylinders of approximately 77-mm length. Substrate temperatures attained during coating runs were predetermined by precisely controlling the gap between the ID of the water-cooled aluminum vacuum chamber body and the OD of the steel substrate.

The tantalum-sputtering target used for this work consisted of a 10-cm length of 9.5-mm OD tantalum rod that was gun drilled to a 3.2-mm ID. The tantalum target was brazed to 9.5-mm OD stainless steel tubing at both ends to allow its extension through the top and bottom of the sputtering apparatus and accommodate connection to cooling water supply and drain lines. The vacuum feed-throughs for the sputtering target contained differentially pumped dual o-ring seals that permitted the target to be moved along the axis of the assembly without breaking vacuum. Thus, a stainless steel portion of the target assembly could act as a "catcher" for material removed during ion cleaning of the steel substrate. Then the tantalum portion of the target could be moved into position for the coating deposit. Test runs were also undertaken in which the target was left in its sputtering position during the substrate ion cleaning process. New targets were presputtered for an hour at 1500 V with krypton prior to being used for coating runs.

For a typical coating run, the apparatus was assembled with the tantalum portion of the target tube below the top level of the lower target shield. The system was helium-leak checked and pumped overnight or longer to a base pressure below 1×10^{-7} Torr using a liquid nitrogen-trapped, oil diffusion-pumped vacuum system. An independent prerun system bakeout was performed to reduce the nonsputtering gas background during the coating phase of the run. The bakeout consisted of a 1-hour filament bake, a gas pressure set (3.0 to 3.5 mTorr krypton), a 30-minute rise to maximum plasma density (12 A plasma current), and a minimum of 1-hour soak at full power. Plasma formation was promoted by application of a 60 V potential between the tantalum ribbon filament and the lid of the apparatus, which operated as the anode. The gas composition in the system during the prerun bakeout and the subsequent coating run was monitored using an independently pumped residual gas analyzer through a variable conductance valve. Between the prerun bakeout and the coating phase of the run, the system was allowed to cool overnight.

For the coating phase of the run, the filament bake, plasma-density rise, and plasma-soak steps were performed as during the prerun system bakeout. Either xenon or argon was substituted for krypton in runs where the sputtering gas variable was being evaluated. Sputtering gas pressures used during the individual runs were adjusted if required to accommodate the system plasma stability for the gas being used. A 10-minute substrate ion cleaning etch (-100 V, 5 mA/cm²) was performed. Minor adjustments were made to the chamber cooling water

temperature during the etch, if needed, to achieve the desired equilibrium substrate temperature. At the end of the substrate cleaning etch, the tantalum target was raised into position, the target voltage was increased to -1500 V, and the plasma current was adjusted to achieve a target current density of 10 mA/cm². A substrate bias of -100 V was maintained throughout the coating run.

Coating Characterization

Optical microscopy was used on cross-sectioned and polished samples to evaluate microstructure and tantalum-phase distribution in the coatings. Knoop microhardness measurements were also performed on the cross-sectioned coating surfaces using a diamond microindentation hardness tester with a 100-g load. X-ray diffraction using copper K-alpha radiation was used to establish the tantalum phases present at the coating growth surfaces.

RESULTS

Tantalum deposits produced using the apparatus and procedures described were generally metallic gray in appearance. Surface finish of the as-deposited coatings varied from being highly reflective for some samples to being quite rough as a result of coating defects protruding from the growth surface. Micrometer measurements taken on the steel cylinder substrates before and after coating indicated that coating thicknesses around the diameter and over the substrate length were uniform to within 10%. Although separate coating adherence tests were not performed on the samples produced, all coatings survived the process of sectioning with a silicon carbide cutoff wheel.

The effect of using the movable target assembly to allow "catching" material removed from the substrate surface during the ion cleaning process was dramatic. Test runs undertaken using a 200°C substrate temperature and krypton-sputtering gas, in which the tantalum target was left in its sputtering position during the substrate ion cleaning process, yielded poor quality coatings characterized by extremely rough surface textures. Micrographs of the cross-sectioned coatings produced in this manner revealed high concentrations of growth defects and a pronounced interfacial layer of beta-phase tantalum (Figure 1). These results compare poorly with the coatings produced under otherwise similar conditions, but with the tantalum portion of the target assembly protected during the substrate cleaning procedure (Figures 2 and 3).

Table 1 presents a summary of sputtering parameters used to produce six samples in which the effects of using different noble gas-sputtering gases were evaluated. Also included in the table are the results of microindentation hardness measurements and growth surface x-ray diffraction analysis of the tantalum coatings. Two samples were produced using each of argon, krypton, and xenon, with other parameters remaining as constant as possible under the constraints of the sputtering apparatus. An effort was made to keep sputtering gas pressures low to minimize gas/sputtered tantalum collisions and hence maximize impact energy. However, the 3 mTorr gas pressure used for xenon and krypton deposits was too low to sustain a stable plasma when using argon, and a system pressure of 5 mTorr was adopted for the argon gas runs. A 200°C substrate temperature was chosen as a target condition for this series of runs.

The tantalum coatings produced using argon as the sputtering gas (samples 121 and 126) were distinct from those produced using krypton and xenon in several regards. First, the coatings were characterized by the presence of two distinct layers (Figure 2), although the form of these layers was not consistent between the two samples. The microhardness values measured

for both sides of the interface in the coating boundary differed somewhat, but were consistently high relative to the microhardness values measured for the krypton- and xenon-produced coatings. Growth surface x-ray diffraction analyses suggested that the growth surfaces of the two samples were distinct in their phase characteristics, one being predominantly beta phase and the other mostly bcc. The coating on sample 121 contained a relatively dark interface layer (~20% of the coating volume) that transitioned irregularly into a lighter-colored surface layer. The thickness of the interface layer varied somewhat with location on the substrate. Typically, the lighter coloration in micrographs of sputtered tantalum coatings is characteristic of the beta phase. Assignment of the outer layer of the 121 coating to the beta phase is consistent with the growth surface x-ray diffraction analysis for this sample and the high microhardness value measured for the outer layer (Table 1).

Both krypton-produced coatings (samples 120 and 132) were predominantly single phase, as indicated by both metallographic analysis (Figure 2) and growth surface x-ray diffraction analysis (Table 1). The coating on sample 132 was characterized by a thin, irregular light-colored interface phase that transitioned to a darker surface phase. This type of phase transition has often been observed in thick tantalum coatings, and has been commonly interpreted as a transition from beta at the interface to a bulk bcc phase. Interpretation of the outer layer as being bcc is consistent with the growth surface x-ray diffraction analysis and the microhardness values obtained for this layer (Table 1). Microhardness measurements taken on the light interface phase were inconsistent due to the small and irregular size of the light-colored features, but were generally much higher (>900 Knoop) than the corresponding values obtained for the darker layer. The second krypton-produced sample (120) showed no indication of a biphasic deposit. All analyses suggested that the coating on sample 120 was 100% alpha phase.

Both samples of xenon-produced tantalum coating (samples 122 and 125) appeared to contain single-phase bcc tantalum (Figure 3). This interpretation is consistent with the results of surface x-ray diffraction and microhardness indentation analyses (Table 1).

Table 2 presents deposition parameters and analytical results for tantalum coatings produced in 25-mm diameter steel cylinders at substrate temperatures ranging from 100°C to 300°C. Figure 3 shows a series of representative micrographs of the cross-sectioned coatings. With the exception of the sample produced at the lowest substrate temperature (sample 138, ~100°C), all of the coatings were either completely or predominantly bcc tantalum. Those samples produced at the intermediate temperatures of ~150°C and ~200°C contained a beta-phase component that appeared either as elongated features growing nearly perpendicular to the coating surface (sample 135) or as the irregular interfacial layer noted above for sample 132. The coatings on the higher-temperature samples (sample 128, 250°C and sample 129, 300°C) appeared to be single-phase bcc tantalum.

DISCUSSION

The results presented here suggest that, within the conditions of our study, the formation of bcc tantalum was promoted by the use of higher atomic weight sputtering gases and elevated substrate temperatures. Coating quality was also improved by the use of a "catcher" rod in place of the tantalum-sputtering target during the ion cleaning of the substrate.

A comparison between the coating structure shown in Figure 1 and those shown in Figures 2 and 3 clearly shows the benefits of eliminating the material from the system that has been removed from the substrate wall during the ion cleaning procedure. Although flat-plate sputtering systems are often configured with a shield or "catcher" plate over the target during substrate ion cleaning, a comparable effect can be difficult to achieve in the cylindrical geometry. Our approach of using the target assembly rod itself as a catcher partially addressed this issue. At best, the collection efficiency on the target assembly rod was relatively poor because of the much smaller area of the rod compared to the surface of the substrate tube (by a factor of ~ 7). Some material was, however, collected on the rod surface during substrate cleaning, and the clean tantalum target surface itself was protected from that material. Thus, oxide and steel removed from the substrate by the ion cleaning was not collected on the target only to be resputtered onto the substrate surface upon initiation of the coating process.

Significant differences in deposition rates, substrate materials, sputtering methods, and sputtering geometries make it difficult to directly correlate our results with those of other researchers who have investigated sputtered-tantalum film-phase characteristics as functions of substrate temperatures and gas-sputtering species. However, our observation that the heavier sputtering gases promote the formation of bcc tantalum is consistent with the work of Ino et al. (ref 19). They demonstrated that the phase characteristics of thin-sputtered tantalum films were related to the energy and/or momentum of ions bombarding the films during growth. Specifically, they found that a stepwise transition from bcc to beta-phase tantalum occurred at characteristic ion bombardment energy or momentum values as those parameters were increased. The ion bombardment energy and momentum required for the bcc to beta-phase transition was shifted to higher values when xenon was used in place of argon as the sputtering gas. Ino et al. attributed the shift in phase characteristics of films grown using different sputtering gases to differences in the abilities of the gases to induce forward recoil-implanted tantalum atom defects into the growing film structures. They also noted that films grown using too little total energy input resulted in poor film quality.

The effect of higher substrate temperatures on the promotion of bcc tantalum over the beta phase in sputtered coatings has generally been attributed to the increased mobility of tantalum atoms in the growing films at the higher temperatures. Substrate temperatures that have been reported as necessary to produce a completely bcc-phase coating using an argon sputtering gas have typically been above 300°C and have been as high as 600°C (ref 15). We speculate that the combination of improved gas purity resulting from system preheating and the use of a "catcher" rod to remove oxide and metallic impurities from initial tantalum deposits allows us to produce single-phase bcc tantalum deposits at substrate temperatures in the 200° to 300°C range. Our demonstration of the ability to produce a fully bcc-phase tantalum coating at temperatures below 300°C using krypton gas and without an intermediate layer brings the required conditions to within the practical realm of gun tube applications.

CONCLUSIONS

The results of this study indicate that elevated substrate temperatures (between 200° and 300°C) and the use of heavier sputtering gases are conducive to the formation of bcc-phase sputtered tantalum on 4340 steel substrates in the cylindrical triode-sputtering geometry. The use of the most common sputtering gas, argon, at a 200°C substrate temperature produced either a coating that was primarily beta-phase or bcc tantalum having unusual hardness characteristics.

When using krypton as the sputtering gas, substrate temperatures in excess of 200°C were required to produce 100% bcc tantalum coatings. The use of a movable target rod assembly allowed ion cleaning of the substrate without deposition of material onto the tantalum target surface and improved the coating microstructure and phase purity.

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Table 1. Effect of Sputtering Gas on the Phase and Microhardness of Thick Tantalum Coatings Produced in a 25-mm Cylindrical Triode Sputtering Apparatus

Run #	Sputtering Gas	Gas Pressure (mTorr)	Substrate Temp. (°C)	Run Duration (hr)	Target Removal Rate (g/A-hr)	Avg. Coating Thickness (μm) ^a	Growth Surface Phase ^b	Coating Microhardness (Avg. Knoop)
121	Argon	5.0	202-210	7.0	5.7	100	Beta	1001/813 ^c
126	Argon	5.0	219-228	9.0	6.3	125	Alpha	923/718 ^c
120	Krypton	3.0	195-219	5.0	9.7	115	Alpha	402
132	Krypton	3.0	203-211	5.7	9.3	135	Alpha	393
122	Xenon	3.0	200-215	2.9	11.6	90	Alpha	341
125	Xenon	3.0	184-188	4.5	12.4	150	Alpha	301

^aAverage coating thickness was calculated based on substrate weight gain and assuming a coating density of bulk tantalum.

^bPredominant phase detected by x-ray diffraction.

^cSamples 121 and 126 contained two distinct layers of tantalum (layer nearest interface/surface layer).

Table 2. Effect of Substrate Temperature on Phase and Microhardness of Thick Tantalum Coatings Produced Using a Krypton Sputtering Atmosphere in a 25-mm Cylindrical Triode Sputtering Apparatus

Run #	Gas Pressure (mTorr)	Substrate Temp. (°C)	Run Duration (hr)	Target Removal (g/A-hr)	Avg. Coating Thickness (μm) ^a	Primary Growth Surface Phase	Coating Microhardness (Avg. Knoop)
138	3.5	96-107	4.9	9.6	125	Beta	1000
135	3.5	149-160	5.0	9.9	135	Alpha	388
132	3.0	203-211	5.7	9.3	135	Alpha	393
128	3.0	250-254	5.0	9.6	130	Alpha	396
129	3.0	292-311	5.0	9.5	125	Alpha	372

^aAverage coating thickness was calculated based on substrate weight gain and assuming a coating density of bulk tantalum.



Figure 1. Photomicrograph of sputtered tantalum coating produced (krypton gas, 200°C substrate temperature) with the tantalum target remaining in sputtering position during substrate ion cleaning.

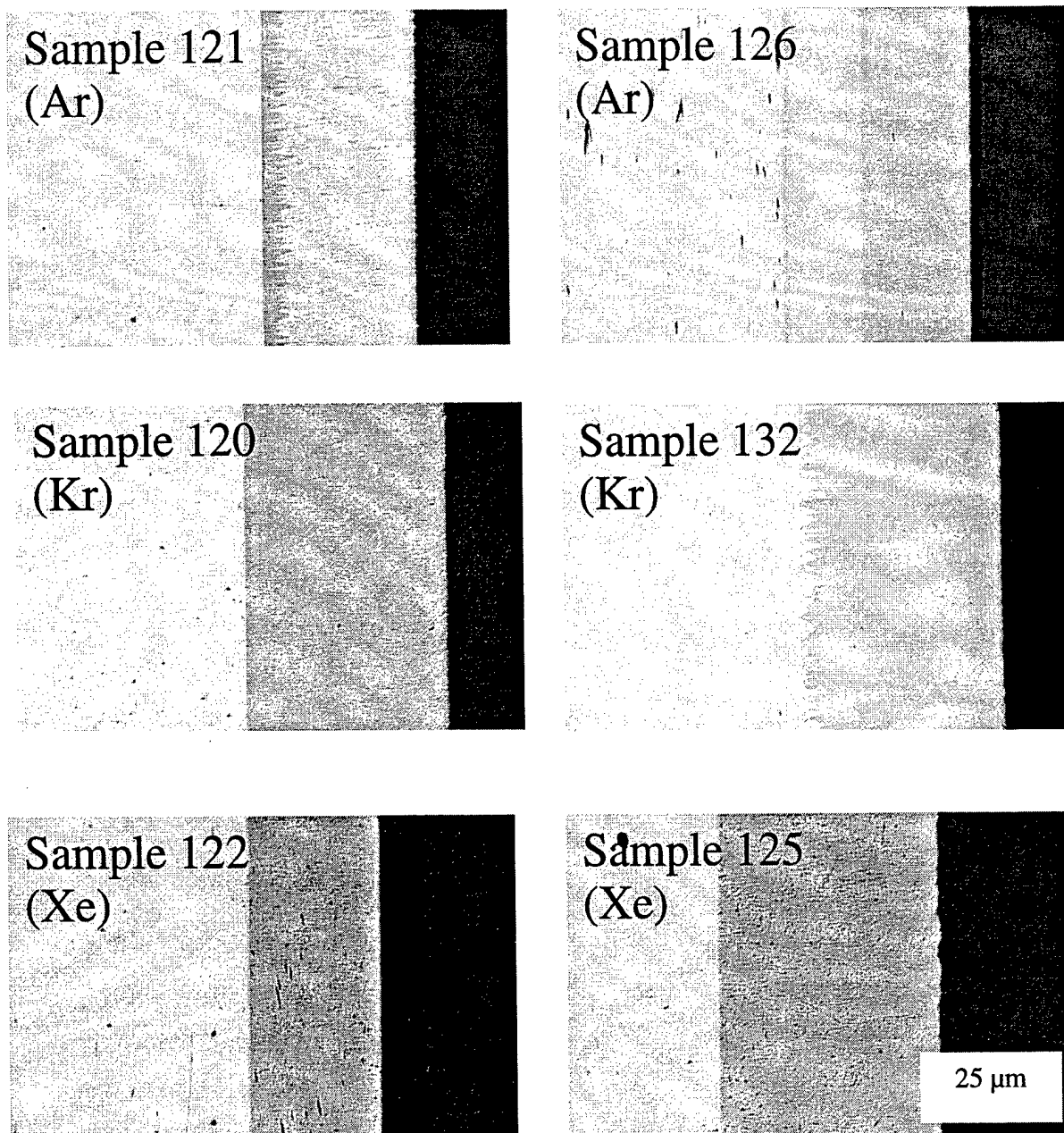


Figure 2. Photomicrographs of sputtered tantalum coatings produced on 4340 steel cylindrical substrates using argon, krypton, and xenon as the sputtering gas. The steel substrate appears as the light-colored area on the left side of each micrograph.

Sample 138
(Kr/100°C)

interface

9

Sample 135
(Kr/150°C)

Sample 132
(Kr/200°C)

Sample 128
(Kr/250°C)

Sample 129
(Kr/300°C)

25 μm

Figure 3. Photomicrographs of sputtered tantalum coatings produced on 4340 steel cylindrical substrates at substrate temperatures from 100°C to 300°C using krypton as the sputtering gas. The steel substrate appears as the light-colored area on the left side of each micrograph.

ENDNOTE

We have noted previously (ref 4) that the beta-phase may be nonuniformly distributed in tantalum coatings coming from different areas of the substrate. Consequently, caution must be exercised when trying to draw conclusions from a single micrograph or from micrographs taken in the same general area of the substrate. Where possible, several different areas were evaluated before choosing the micrographs shown in Figures 2 and 3 as being generally representative of the coating.

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